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IS 13841 (1993): 5-Sulfoanthranilic Acid, Technical [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]

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भारतीय मानक

(Reaffirmed 2002)

5-सल्फोएंथ्रेनिलिक अम्ल, तकनीकी – विशिष्टि

Indian Standard

5-SULFOANTHRANILIC ACID, TECHNICAL — SPECIFICATION

UDC 667·285·1

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

August 1993

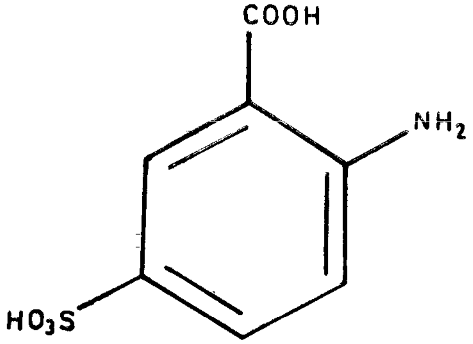
Price Group 3

Dye Intermediates Sectional Committee, PCD 11

FOREWORD

This standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

5-Sulfoanthranilic acid ($C_7 H_7 NO_5 S$) is used as an intermediate for reactive dyes. It is also known as 2-amino-5-sulfobenzoic acid. It is represented by the following structural formula:



5-Sulfoanthranilic Acid
(Molecular Mass 217)
CAS Registry Number [3577-63-7]

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

5-SULFOANTHRANILIC ACID, TECHNICAL — SPECIFICATION

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for 5-sulfoanthranilic acid, technical.

2 REFERENCES

The following Indian Standards are necessary adjuncts to this standard:

IS No.	*Title*
1070 : 1992	Water for general laboratory use (*third revision*)
2552 : 1989	Steel drums (galvanized and ungalvanized) (*third revision*)
5299 : 1969	Methods of sampling and tests for dye intermediates

3 REQUIREMENTS

3.1 Description

The material shall be moist, yellow to yellow brown in colour.

3.2 The material shall also comply with the requirements given in Table 1.

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in steel drums (*see* IS 2552 : 1989) lined with suitable polyethylene film or as agreed to between the purchaser and the supplier. Each container shall be securely closed.

4.2 Marking

Each container shall legibly and indelibly bear the following information:

a) Name of the material;
b) Indication of the source of manufacture;
c) Tare, net and gross mass;
d) Batch number; and
e) Month and year of manufacture.

4.2.1 The containers may also be marked with the Standard Mark.

5 SAMPLING

5.1 Representative samples of the material shall be drawn as prescribed in **3** of IS 5299 : 1969.

5.2 Number of Tests

5.2.1 Tests for assay and determination of anthranilic acid shall be conducted on each of the individual samples.

5.2.2 Tests for the determination of remaining characteristics, namely, description, matter insoluble in dilute sodium carbonate solution and acid other than 5-sulfoanthranilic acid shall be conducted on the composite sample.

5.3 Criteria for Conformity

5.3.1 *For Individual Samples*

The lot shall be declared as conforming to the requirements of assay and anthranilic acid content if each of the individual test results satisfies the relevant requirement given in Table 1.

Table 1 Requirements for 5-Sulfoanthranilic Acid, Technical

(*Clauses* 3.2)

Sl No.	Characteristic	Requirements	Methods of Test, Ref to Annex
(1)	(2)	(3)	(4)
i)	Matter insoluble in dilute sodium carbonate solution, percent by mass, *Max*	0·20	A
ii)	Assay, percent by mass, *Min*	60·0	B
iii)	Acid (as HCl) other than 5-sulfoanthranilic acid (on 100 percent basis), percent by mass, *Max*	8·0	C
iv)	Anthranilic acid (on 100 percent basis), percent by mass, *Max*	1·0	D

5.3.2 *For Composite Sample*

For declaring the conformity of the lot to the requirements of all other characteristics (*see* **5.2.2**) tested on the composite sample, the test results for each of the characteristics shall satisfy the relevant requirements given in **3.1** and Table 1.

6 TEST METHODS

6.1 Tests shall be carried out according to the methods prescribed in Annexes, as indicated in col 4 of Table 1.

6.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* 1070 : 1992) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

[*Table* 1, *Sl No.* (i)]

DETERMINATION OF MATTER INSOLUBLE IN DILUTE SODIUM CARBONATE SOLUTION

A-1 APPARATUS

A-1.1 Weighing Bottle

A-1.2 Beaker — 1 litre.

A-1.3 Sintered Glass Crucible — G 3.

A-1.4 Buchner Funnel

A-2 REAGENTS

A-2.1 Sodium Carbonate Solution — 10 percent.

A-2.2 Brilliant Yellow Paper

A-3 PROCEDURE

Weigh accurately about 20·0 g of the sample and transfer into a 1-litre beaker. Add 600 ml hot water followed by sodium carbonate solution (**A-2.1**) till faint alkaline test on brilliant yellow paper. Heat to dissolve completely. Filter hot through sintered glass crucible using Buchner funnel and wash with hot water till free from alkali. Preserve the filtrate and washings for assay (*see* Annex B). Dry the residue at 100°C to a constant mass.

A-4 CALCULATION

$$\text{Matter insoluble in dilute sodium carbonate solution, percent by mass} = \frac{M_1 \times 100}{M}$$

where

M_1 = mass in g of residue, and

M = mass in g of sample taken for the test.

ANNEX B

[*Table* 1, *Sl No.* (ii)]

ASSAY

B-1 APPARATUS

B-1.1 Pipette — 50 ml.

B-1.2 Beaker — 1 litre.

B-1.3 Volumetric Flask — 1 litre.

B-2 REAGENTS

B-2.1 Concentrated Hydrochloric Acid

B-2.2 Sodium Nitrite Solution — 0.1 N.

B-2.3 Potassium Starch Iodide Paper

B-3 PROCEDURE

Cool the filtrate and washings (**A-3**) to room temperature. Dilute to 1 000 ml with water in a volumetric flask and mix well. Pipette out 50 ml aliquot in a 1-litre beaker. Add 400 ml ice-cold water followed by 35 ml of concentrated hydrochloric acid and titrate while stirring mechanically at 15-20°C with standard sodium nitrite solution (**A-2.2**) using potassium starch iodide papers to a 5 minute end point.

B-4 CALCULATION

$$\text{Assay, percent by mass} = \frac{V \times N \times 217}{M}$$

where

V = volume in ml of sodium nitrite solution,

N = normality of standard sodium nitrite solution, and

M = mass in g of sample taken for the test.

ANNEX C

[*Table* 1, *Sl No.* (iii)]

DETERMINATION OF ACID OTHER THAN 5-SULFOANTHRANILIC ACID (as HCl)

C-1 APPARATUS

C-1.1 Conical Flask — 500-ml.

C-2 REAGENTS

C-2.1 Ethyl Alcohol — neutral.

C-2.2 Standard Sodium Hydroxide Solution — N/1.

C-2.3 Phenolphthalein Indicator

C-3 PROCEDURE

Weigh accurately about 5·0 g of sample in a 500-ml conical flask, add 50 ml distilled water and 250 ml neutral ethyl alcohol. Warm to dissolve completely. Titrate with standard sodium hydroxide solution (**C-2.2**) using phenolphthalein indicator.

C-4 CALCULATION

$$\text{Total acidity as sulfoanthranilic acid (as HCl), percent by mass } X = \frac{V \times N \times 10{\cdot}86}{M}$$

$$\text{Acid other than sulfoanthranilic acid (as HCl), (on 100 percent basis), percent by mass} = \frac{(X - A) \times 33{\cdot}6}{A}$$

where

V = volume in ml of standard sodium hydroxide solution;

N = normality of standard sodium hydroxide solution;

M = mass in g of sample taken for the test; and

A = percent assay (*see* **B-4**).

ANNEX D

[*Table* 1, *Sl No.* (iv)]

DETERMINATION OF ANTHRANILIC ACID

D-1 OUTLINE OF THE METHOD

Paper chromatographic method is prescribed for determination of anthranilic acid in 5-sulfoanthranilic acid.

D-2 APPARATUS

D-2.1 Chromatographic Tank

D-2.2 Sprayer

D-2.3 Ultra Violet Lamp

D-3 REAGENTS

D-3.1 Ammonium Hydroxide Solution — 2 percent.

D-3.2 Anthranilic Acid

D-3.3 n-propanol : Water — 70 : 30.

D-3.4 Sodium Nitrite Solution — 1 N.

D-3.5 Hydrochloric Acid — 1 M.

D-3.6 Whatman Paper — 3 mm size suitable for paper chromatography.

D-3.7 Chromotropic Acid — 0·05 percent.

D-3.8 Sodium Acetate Solution — 40 percent.

D-4 PREPARATION OF SOLUTIONS

D-4.1 Standard Sample Solution

Weigh accurately 2·0 g of the material in ammonia solution (**D-3.1**) and make up to 100 ml with distilled water.

D-4.2 Standard Anthranilic Acid

Weigh accurately 0·02 g of Anthranilic acid and dissolve in ammonia solution (**D-3.1**). Make up to 100 ml.

D-5 PREPARATION OF TANK

D-5.1 Place 200 ml of developing mixture (**D-3.3**) into the chromatographic tank and replace the lid. Allow the equilibrate for about an hour.

D-6 DEVELOPMENT OF CHROMATOGRAM

D-6.1 Spot 10 μl of sample solution (**D-4.1**) and 10 μl of anthranilic acid solution (**D-4.2**) on the paper and suspend the paper from the supporting rods by means of clips so that the lower edge of the paper is just touching the surface of the developing solvent but is not supported on the bottom of the tank. Allow the chromatogram to develop for about 15 hours. After developing and drying at 100°C for 10 minutes. Develop the flourescence by holding the paper over a beaker containing concentrated ammonium hydroxide solution. Examine the paper under UV lamp for characteristic flourescence. A typical chromatogram so developed is given in Fig. 1.

D-6.1.1 Diazotize the paper in nitrous acid fumes by holding the paper over a beaker containing sodium nitrite solution and hydrochloric acid. Remove the fume and couple the chromatogram by spraying chromotropic acid (**D-3.7**) and sodium acetate solution (**D-3.8**) (1 : 1). Compare the standard spot and evaluate.

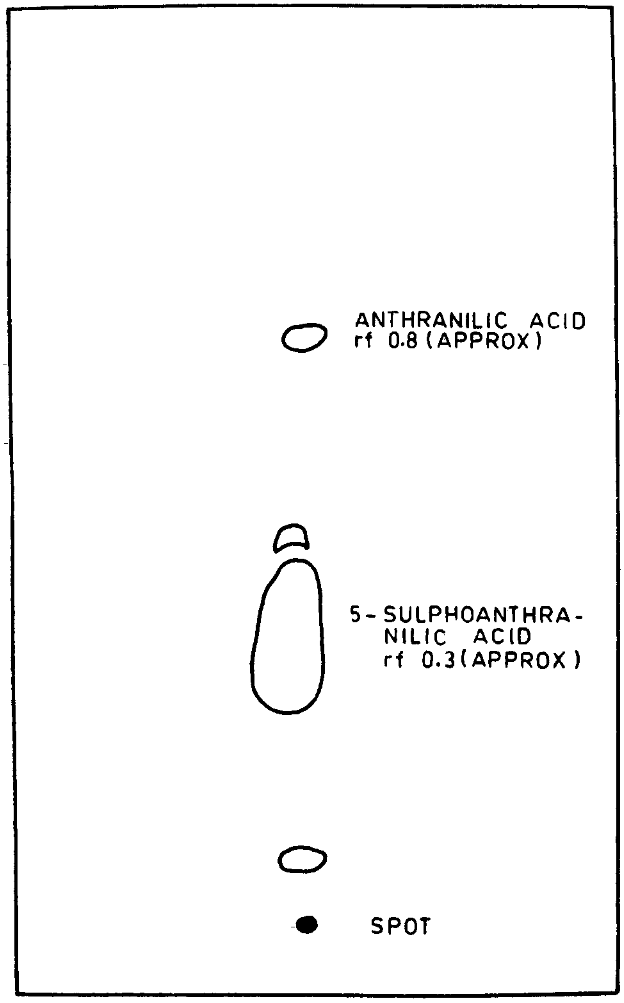


FIG. 1 A TYPICAL CHROMATOGRAM FOR ESTIMATION FOR ANTHRANILIC ACID IN 5-SULFOANTHRANILIC ACID

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